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1,3-Diethyl-1,1,3,3-tetraphenyldisiloxane

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Key indicators

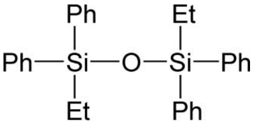
Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.157
Data-to-parameter ratio = 10.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Molecules of the title compound, $C_{28}H_{30}OSi_2$, are centrosymmetric, with the O atom lying on an inversion centre. The ethyl substituents are placed *trans* along the strictly linear Si—O—Si fragment.

Comment

Crystal structures of *ca* 40 acyclic disiloxanes are known, but a sound explanation why their Si—O—Si angles vary within the 140–180° range still cannot be provided. The Cambridge Sructural Database (CSD, Version 5.27 of 2006 with updates; Allen, 2002) stores data for eight symmetric disiloxanes of formula $Ph_2RSiOSiRPh_2$; these include R = Me (CSD refcode GIMZUX; Wojnowski *et al.*, 1988), ^{t}Bu (CSD refcode DEPGOU; Karle *et al.*, 1986) and Ph [CSD refcodes OXTPSI02 (Kooijman *et al.*, 1999) and OXTPSI10 (Glidewell & Liles, 1978)], for which the Si—O—Si angles are 140, 152.5 and 180°, respectively. We have prepared and structurally characterized 1,3-diethyl-1,1,3,3-tetraphenyldisiloxane ($Ph_2EtSiOSiEtPh_2$), (I), a compound which fits well in this series. The structure of (I) is shown in Fig. 1 and selected geometric parameters are given in Table 1.



$\mathrm{Ph{-}Si(Ph)(Et){-}O{-}Si(Et)(Ph){-}Ph}$

(I)

The two halves of the molecule of (I) are related by an inversion centre located at atom O1. It follows that, despite similarities between alkyl substituents (Me, Et and ^{t}Bu), the geometry of (I) resembles most closely that of hexaphenyldisiloxane; in both compounds (R = Et, Ph), the central Si—O—Si fragment is strictly linear. The same holds for Si—O bond lengths which are virtually the same in (I) and $Ph_3SiOSiPh_3$ [1.616 (1) Å according to Glidewell & Liles (1978), and 1.620 (1) Å determined at 150 K by Kooijman *et al.* (1999)], but remarkably longer in the case of $Ph_2MeSiOSiMePh_2$ [1.637 (2) and 1.640 (2) Å; Wojnowski *et al.*, 1988] and $Ph_2{}^tBuSiOSi{}^tBuPh_2$ [1.633 (1) and 1.638 (1) Å; Karle *et al.*, 1986]. These two sets of observed values correlate with the type of disiloxane structure, linear or bent, and the mutual arrangement of the alkyl substituents. Unlike $Ph_2EtSiOSiEtPh_2$, (I), where the ethyl groups are *trans*, in $Ph_2MeSiOSiMePh_2$ and $Ph_2{}^tBuSiOSi{}^tBuPh_2$ the alkyl substituents are *gauche* along the Si···Si axis (see Newman projections in Fig. 2). This conformation is accompanied by a bending of the Si—O—Si fragment and elongation of the Si—O bonds. The

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same phenomenon is observed in other known disiloxanes of type $(Ph_2RSi)_2O$ [CSD refcodes DAPNAK and DAPNOY (Nitsche *et al.*, 2003), TANKOJ (Lee *et al.*, 2002), ECOHUA and EDAFAR (Dirnens *et al.*, 2003)]; only those with *R* substituents in *trans* positions have linear Si—O—Si skeletons. The case of 1,3-bis[3-(3-pyridyl)isoxazolin-5-yl]-1,1,3,3-tetraphenyldisiloxane (Dirnens *et al.*, 2003) is especially interesting as all three types of conformers are known for this compound. The *trans* form has a linear skeleton, but the *cis* and *gauche* forms are bent.

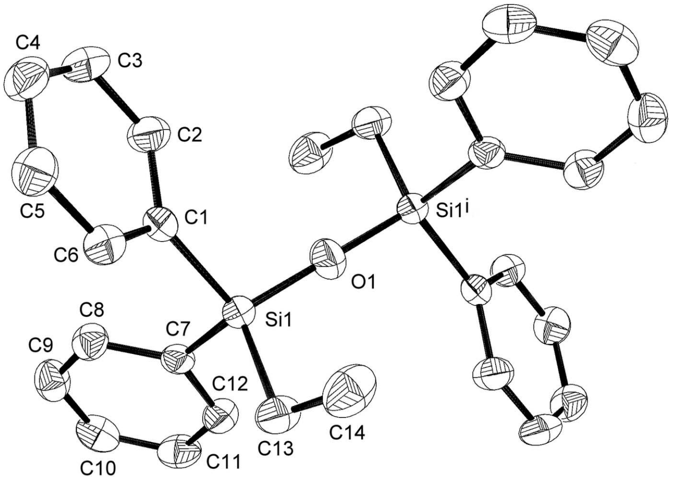

Figure 1
The molecular structure and atom-numbering scheme for (I), with displacement ellipsoids drawn at the 50% probablilty level. H atoms have been omitted for clarity. [Symmetry code: (i) $-x + 2, -y + 1, -z + 1$.]

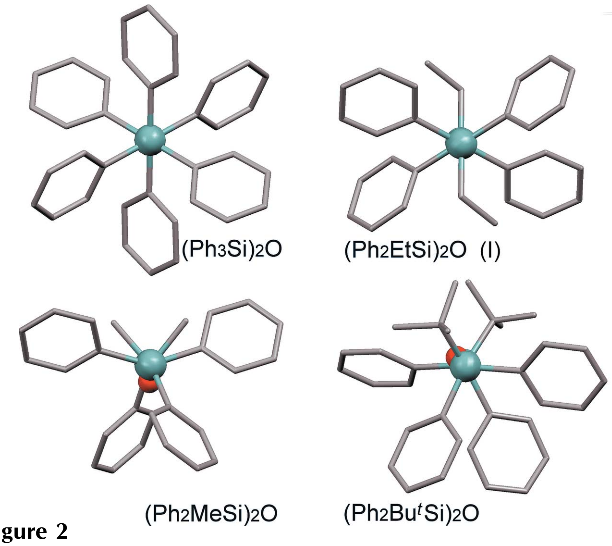

Figure 2
Newman projections along the Si···Si axis for $(Ph_2RSi)_2O$ molecules (R = Me, Et, ^{t}Bu and Ph).

Experimental

Crystals of (I) suitable for X-ray measurements were obtained by hydrolysis of a toluene solution of 1,3-diethyl-1,1,3,3-tetraphenyldisilthiane (Becker & Wojnowski, 1982) with an excess of aqueous $AgNO_3$, followed by the usual workup and crystallization of the crude product from hexane–benzene (4:1 *v*/*v*).

Crystal data

$C_{28}H_{30}OSi_2$
$M_r = 438.7$
Monoclinic, $P2_1/c$
$a = 7.8479\ (14)$ Å
$b = 20.603\ (3)$ Å
$c = 7.5978\ (15)$ Å
$\beta = 98.541\ (19)^\circ$
$V = 1214.9\ (4)$ Å^3
$Z = 2$
$D_x = 1.199$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.16$ mm^{-1}
$T = 200\ (2)$ K
Block, colourless
0.22 × 0.20 × 0.16 mm

Data collection

Kuma KM-4-CCD diffractometer
ω scans
Absorption correction: none
4916 measured reflections
2028 independent reflections
1590 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.055$
$\theta_{\rm max} = 25^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.157$
$S = 1.14$
2028 reflections
203 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0917P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.35$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.39$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.023 (6)

Table 1
Selected geometric parameters (Å, °).

Si1—O1	1.6181 (7)	Si1—C1	1.867 (3)
Si1—C13	1.857 (3)	Si1—C7	1.870 (3)
O1—Si1—C13	109.29 (11)	Si1—O1—Si1^i	180
O1—Si1—C1	108.74 (9)	C2—C1—Si1	120.4 (2)
C13—Si1—C1	112.27 (14)	C6—C1—Si1	122.4 (2)
O1—Si1—C7	108.48 (9)	C8—C7—Si1	123.3 (2)
C13—Si1—C7	108.01 (13)	C12—C7—Si1	120.1 (2)
C1—Si1—C7	109.98 (12)	C14—C13—Si1	115.6 (2)

Symmetry code: (i) $-x+2, -y+1, -z+1$.

All H atoms were found in an electron difference Fourier map and refined without constraints or restraints.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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